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OIL-NICOTINE, A PROMISING NEW INSECTICIDE

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Oil-Nicotine, A Promising New Insecticide*

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Poisons for the control of insect pests fall into two main groups, the stomach poisons for chewing insects and the contact poisons for sucking insects. The most satisfactory stomach poisons are the arsenates of lead, calcium and magnesium, and certain fluorine compounds, applied as dusts and aqueous sprays. The most satisfactory contact poisons are nicotine sulfate, oil emulsions and lime-sulfur, applied in aqueous solution.

The arsenical and fluorine insecticides are poisonous to man, and this has led to the establishment of rigorous requirements by the federal government in regard to the amount of arsenical, lead, or fluorine residues which may be present on foods offered for sale in interstate commerce. The prospect of even smaller tolerances in the near future and the need for new insecticides less toxic to man have led to a nationwide study by chemists and entomologists of many possible compounds. As a result, several promising synthetic and naturally occurring organic compounds, such as certain thiocyanates, pyrethrum, rotenone, phenothiazine, and cyclohexyl-phenol, have been developed. Several novel methods of application have also been made available. One contribution from this Station is the development of oil-nicotine as a horticultural spray and its application by atomization. Because this new spray material shows promise of combining the merits of both contact and stomach poisons for a number of noxious insects, a preliminary account is given at this time.

HISTORY OF FORTIFIED ATOMIZED OILS

The use of atomized oils, fortified with pyrethrum or other toxic substances, as contact insecticides, is a recent development in eco-

* Preliminary report on a joint project of the Department of Entomology and Botany and the Department of Chemistry.

nomic entomology. As early as 1903, Volck (17) in California reported trying the application of oil by the use of an air blast. However, it was not until 1933, by the work of various California entomologists and Ginsburg (2, 6, 9, and 12) of New Jersey, that the efficacy of oil-fog sprays for horticultural purposes was shown. Up to the present, the main toxicant employed was pyrethrum of which the components of insecticidal value are Pyrethrins I and II. This material was incorporated into highly refined petroleum base oil by steeping the ground pyrethrum flowers in the oil or by adding a concentrated pyrethrum extract to the oil by the use of intermediate solvents. Recently, rotenone incorporated in oil has been tested by a number of workers and a patent for its incorporation was assigned to one of the pioneer companies in the development of household pyrethrum-oil sprays.

As early as 1929, R. H. Smith (15) of California reported adding nicotine to spray oil in which he claimed it was only very slightly soluble. He found a few suitable intermediate solvents, among which was butyl alcohol. All his efforts to fix nicotine permanently in oil failed.

In 1933, Le Pelley (11) reported attempting to extract the toxic principles of tobacco in oil but did not work out a satisfactory method of procedure. Also, in 1933, Herbert (9) published an account of the use of a light oil containing pyrethrum and nicotine, for spraying with airplane equipment.

Smith, Meyer and Persing (16), in 1934, used 95 percent free nicotine, dissolved in kerosene, gasoline and petroleum ether, plus alcohol as an intermediate solvent, as an atomized spray for the control of codling moth. They concluded that the material applied in that manner was impractical and turned their attention to a "Nicofumer" used to vaporize atomized free nicotine or nicotine sulfate in a portable fumatorium by means of pressure and heat.

The use of oil-nicotine as a horticultural spray was investigated in 1936 at the Kentucky Agricultural Experiment Station in a cooperative project between the Department of Entomology and Botany and the Department of Chemistry (14).

PREPARATION OF OIL-NICOTINE SPRAY

Ninety-five percent free nicotine held in a completely volatile, highly refined hydrocarbon distillate¹ by ethylene dichloride was used in the first tests. This mixture was homogeneous when first prepared but at the end of two days, in a 5 percent solution, brown droplets composed principally of water with some other impurities appeared on the bottom of the flask. One percent and 2.5 percent solutions of nicotine in the base oil, using 95 percent free nicotine and ethylene dichloride, remained stable for at least 10 days; after that time water and other impurities began to settle out.

It was found that 100 percent free nicotine was entirely soluble in several light, highly refined oils without the use of an intermediate solvent. The resulting solution was clear and appeared to be permanent. Commercial 95 and 50 percent free nicotine can also be used in the preparation of an oil-nicotine spray. When a sample of commercial 95 percent nicotine (by analysis 92.2 and 92.4 percent) was shaken with sufficient oil to yield a 1 per cent solution, a slightly cloudy mixture was formed. This was centrifuged and the clear, water-white solution of nicotine in oil separated from the brown aqueous deposit. The oil-insoluble material was found by analysis to contain only .119 percent of the total nicotine. The oil, then, by difference, contained .9988 per cent nicotine.

The base oil also took up almost all the nicotine from a 50 percent free nicotine (49.5 percent by analysis). The aqueous layer that separated after shaking contained 1.49 percent of the total nicotine, giving a .985 percent solution of nicotine in oil.

It appears from these results that the loss of nicotine in mixing small amounts of spray material is negligible when 95 and 50 percent free nicotine are used as sources of nicotine. The correct amount of 40 percent, 50 percent, or 95 percent free nicotine may be shaken in one container with the correct amount of oil, allowed to settle, and the resulting solution poured into the atomizer spray

¹ The highly refined hydrocarbon distillate used in these tests will be referred to hereafter as the base oil. Unless otherwise mentioned, it has the following specifications:

Specific gravity	.775-.785
Saybolt viscosity at 100° F.	30 secs.
Flash point	170-180° F.
Initial boiling point	370-390° F.
Distillation end point	480-500° F.
Unsulphonatable residue	Above 98%
Color	Water white
Odor	Practically none

container. The dark residue left in the bottom of the first container may be discarded.

TOXICITY OF OIL-NICOTINE TO INSECTS

The first work with oil-nicotine, as mentioned above, was done with 95 percent free nicotine held in base oil by means of ethylene dichloride. It was found that nicotine in oil is much more toxic to insects than are comparable aqueous nicotine solutions. This is undoubtedly true because of the penetrating action of the oil and some additive toxic effect of the oil itself. A comparison of nicotine in oil and nicotine in water is given in table 1. The material was applied to adult green June beetles with a Devilbiss No. 16 atomizer.

Table 1. Relative toxicity of oil-nicotine and aqueous nicotine sprays applied to adult green June beetles.

Spray material	Percent killed	Number of insects	Number of tests
5% nicotine in water with spreader	none	15	3
2½% nicotine in water with spreader	none	15	3
5% nicotine in oil	100	40	8
2½% nicotine in oil	80	15	3
1½% nicotine in oil	40	15	3
5% nicotine in oil	7	15	3
Oil only	none	15	3

Later, laboratory tests of oil-nicotine were made, using base oil to which 100 percent free nicotine had been added directly. The results of these tests and of others made earlier with ethylene dichloride as an intermediate solvent for 95 percent free nicotine, are given in tables 2 and 3. In some tests oil-nicotine was compared in toxicity with the base oil containing pyrethrum and rotenone, all containing equal amounts of the same intermediate solvent. In some, a modification of the apparatus described by Campbell and Sullivan (3) was used; in others, use was made of a small, continuous hand atomizer designed by Ritcher and Calfee.

In interpreting these results it should be remembered that only comparative toxicity between the materials is shown in the results of the bell-jar-settling mist tests, not the complete effectiveness of any material. The hand-sprayer tests more nearly approximate field conditions but here, too, an attempt was made to apply the materials in small enough amount to give low kills in the oil checks. It is of interest that nicotine in oil exhibited a spectacular knock-down that was much quicker than any other toxicant used.

Table 2. Relative toxicity of oil-nicotine, oil-pyrethrum and oil-rotenone, applied as a mist to insects under a bell jar.

Treatment	Imported cabbage worm	Percent killed Pea aphis	Nasturtium aphis	Red spider
Oil-nicotine 2 percent	42	—	—	—
Oil-nicotine 1 percent	20	53	95	94
Oil-pyrethrum 0.2 percent	87	54	86	93
Oil-pyrethrum 0.1 percent	78	—	—	—
Oil-rotenone 0.2 percent	—	100	—	—
Oil only	17	6	19	—
Nothing	—	0	0	—

Table 3. Relative toxicity of oil-nicotine, oil-pyrethrum and oil-rotenone, applied to insects by means of a hand atomizer. Percent killed.*

Treatment	Cabbage looper	Southern cabbage worm	Imported cabbage worm	Tingids	Ham mite	Apple leaf hopper nymphs	Potato beetle	Mealy bug
Oil-nicotine 3%	98	—	—	—	100	—	—	—
Oil-nicotine 2½%	—	—	—	—	—	—	—	100
Oil-nicotine 2%	—	—	—	—	97	—	—	—
Oil-nicotine 1%	71	75	77	95	71	95	33	—
Oil-nicotine 0.63%	—	—	—	—	—	97	—	—
Oil-pyrethrum 0.2%	—	—	—	98	—	—	—	—
Oil-pyrethrum 0.1%	89	78	—	—	—	—	—	—
Oil-pyrethrum 0.05% ..	83	62	97	—	—	—	—	—
Oil-rotenone 0.2%	—	—	—	62	—	—	—	—
Oil only	28	18	—	64	70	31	—	—
Nothing	—	2	—	—	78	—	—	—

* See appendix for results in detail.

It is concluded from the tests reported in these tables and from field and greenhouse observations that nicotine in oil gives a high per cent kill of many kinds of insects. It also appears that 1 percent free nicotine in oil is about equivalent in toxicity to .1 percent pyrethrum in oil. Double this amount of pyrethrum was used in some tests but this strength was probably unnecessary since various workers agree that pyrethrum reaches its maximum toxicity at about .1 percent. One percent free nicotine in oil is about the right concentration for field work.

Other insects on which good kills were had with nicotine in oil are adult white flies, squash bug nymphs, adult blister beetles, thrips, stink bugs, and a number of common household and stored-products pests. The control obtained on mealy bugs, is illustrated by Figure 1. Both pots of lilies were originally heavily infested. The plant on the left was atomized with 1 percent nicotine in oil.

EFFECT ON PLANTS

Ginsburg (6, 7, 8), of New Jersey, and others (1) have shown that certain highly refined, volatile hydrocarbon distillates, alone and containing pyrethrum, may be safely applied to plants in the form of a fog. The writers found that free nicotine in similar oils also may be safely applied to plants, provided use is made of proper equipment. The plants to which nicotine in oil was applied as a fog without causing appreciable injury are apple, peach, grape, elm, lettuce, tomato, bean, radish, cabbage, turnip, sweetpotato, tobacco, red clover, morning-glory, nasturtium, marigold, zinnia, snapdragon, cosmos, rose, geranium, arbor vitae, and Scotch pine. Of these, lettuce, bean, sweetpotato, and clover are especially sensitive to overdose.



FIGURE 1. Mealy-bug infested lilies. The plant on the left was atomized with 1 percent nicotine in oil.

Since the oil used in these tests is highly refined and completely volatile, it is much safer on plants than less refined oils. Some hardy plants can be drenched with it and show no resulting injury. According to Ginsburg (7), chrysanthemums can be dipped in the oil without injury. The effect on foliage of applying 2.5 percent oil-nicotine with a semi-coarse continuous sprayer is shown in table 4.

Table 4. Comparative effect of oil-nicotine applied to plants with an atomizer and with a semi-coarse sprayer.

	Injury after spraying					
	With atomizer			With semi-coarse sprayer		
	24 hrs. after	7 days after	14 days after	24 hrs. after	7 days after	14 days after
Apple	0	0	0	0	xx	xx
Peach	0	0	0	0	xx	xx
Grape	0	0	0	x	xxx	xxx
Lettuce	0	0	0	xxx	xxx	xxx
Tomato	0	0	0	xxx	xxx	xxx
Bean	x	x	x	x	xxx	xxx
Radish	0	0	0	xxx	xxxx	dead
Red clover	0	0	0	x	xx	xxx
Nasturtium	0	0	0	xxx	xxx	dead

Legend: 0, no injury; x, very slight; xx, slight; xxx, considerable; xxxx, much.

Two types of burn are noticeable when excessive nicotine in oil is applied to foliage. The more usual type is where dead spots and blotches develop on the leaves wherever oil has penetrated. This injury can be detected almost immediately after treatment. The second type is an insidious one resulting from overdosing hardier plants or application at too low temperature for rapid volatilization. It is characterized by a loss of greenness and gradual yellowing of foliage. Such injury was found on drenched tobacco and lilies.

EFFECT ON ANIMALS

It is well known that nicotine is very toxic to warm-blooded animals.* Inhaled in extremely small quantity, it is a throat irritant and causes coughing. As much as 5 percent free nicotine in base

* Not only are nicotine and its salts deadly poisons, taken internally, but they may produce serious poisoning if they come in contact with the skin. If a strong solution of nicotine is spilled on the skin, it should be washed off immediately with water. Oil-nicotine should be wiped off immediately and the skin washed with soap and water.

oil was applied to plants, where there was adequate ventilation, without much discomfort to the operator. Against most insects, 1 percent free nicotine in oil is sufficient. A fog of this strength may cause slight throat irritation and headache if one remains in a closed greenhouse with the fumes for more than an hour. One percent can be used outdoors with impunity and also in the greenhouse if there is enough ventilation. Where strong dosages are used or where prolonged breathing is necessary, an effective mask for the nose may be made from several thicknesses of dampened cheesecloth. It is possible to mask the nicotine odor with pine tar or creosote odors but they do not prevent the throat irritation common to all nicotine sprays. One percent free nicotine in oil seemed less offensive to some observers than a 1:800 aqueous nicotine sulfate spray.

APPARATUS FOR APPLYING OIL-NICOTINE

Up to the present, use has been made of airplane equipment, blower vaporizers, compressed air and knapsack sprayers, for the application of atomized oils (4 and 5). Ginsburg (8) used a fog sprayer in many of his tests. All this equipment is available commercially only to a limited extent, the prices asked are excessive and the equipment is not entirely satisfactory.

It was found here that for small-scale use, a continuous atomizer (Fig. 2) was much more satisfactory than a continuous sprayer of the type used by Ginsburg. This atomizer does not drip and gives a steady flow of extremely small droplets, with little effort in pumping. Inasmuch as this continuous atomizer should have immediate commercial possibilities for household and garden use, it is described in some detail. Its construction is shown in Fig. 3. F represents the barrel, plunger and valve taken from an ordinary continuous sprayer. A is a metal air chamber permanently fixed to the barrel. The liquid reservoir B is attached inside A. There is no communication between the two. A small hole in the filling cap, E, subjects the liquid to atmospheric pressure. C is a copper tube leading from the bottom of the liquid-container to the air nozzle D. The orifices in both C and D are approximately .75 mm in diameter. C projects slightly past the perimeter of the opening D.

In operation, the forward action of the plunger forces part of the displaced air thru the opening D and compresses the rest in

chamber A, building up the pressure there. This pressure reserve, with the closing of the valve, causes a flow of air to continue thru D on the return stroke of the plunger. The continuous stream of air from A across C causes a lower atmospheric pressure in C, effect-

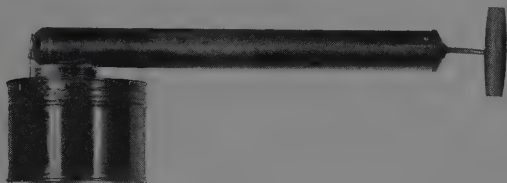


FIGURE 2. New continuous hand atomizer for the application of atomized oil to plants.

ing a rise of liquid to the tip. The stream of air at high velocity, passing the opening of C breaks up minute quantities of liquid into a fine, penetrating fog.

Small 2- to 4-gallon continuous sprayers for use in the garden

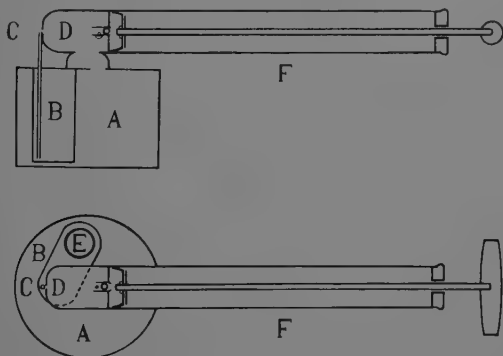


FIGURE 3. Construction of new continuous hand atomizer. A, air chamber; B, liquid container; C, liquid-delivery tube; D, air nozzle; E, filling cap; F, pump barrel.

may be easily adapted for use with atomized oils. An atomizer nozzle and liquid container may be screwed on the end of the spray rod and the 2- to 4-gallon tank used as an air-pressure tank. One pumping up of such a 3-gallon continuous atomizer was sufficient for 10 to 12 minutes operation.

For large-scale work, such as in greenhouses, a nozzle is needed

that produces a large volume of fog and still breaks up the oil into fine droplets. Several nozzles for this purpose, built on the principle of the Venturi tube, have been designed and used with both compressed air and steam. The new atomizing gun, the construction of which is shown in Fig. 4, is a device for greenhouse use.

In Fig. 4, A is an assembly drawing with parts as follows: 1. Cap nut, taper-drilled to form a tapered tube. 2. Short brass tube projected into the tapered tube. 3. Threaded section, brazed or threaded to 2, upon which 1 operates for adjustment of the Venturi effect. 4. Compression spring for holding the adjustment. 5. (In cross-section) Shows drillings for passage of air. 6 Small flexible metal tube, attached to 2 and 8 by either brazed or threaded unions. 7. Larger flexible metal tube similarly attached to 3 and to 11. 8. Oil-insecticide feed tube brazed or cast to 11 at point of emergence. 9. Screw cap to fit a standard can or glass container, either cast with or brazed to 11. 10. Insecticide container. 11. An aluminium or alloyed casting consisting of a short barrel, handle, and accessory parts as indicated. 12. Combination of air valve and air adjustment, trigger and accessory parts. 13. Air passage to the valve drilled in the casting 11. 14. Adapter either threaded or flared for the attachment of an air hose to a source of compressed air.

Operation. The compressed spring (d) exerts a force between (c) and (e) that holds the needle (g) tight against the valve seat (a). Rotation of (g) changes the position of (k), (i), and (j) which regulates the extent to which the trigger may be retracted and consequently the open position of (g) and (a).

C is a cross section of 11, the casting.

(a) Air chamber around the needle. (b) Drilled air tube offset to pass the trigger mechanism. (c) Slot in which the trigger operates.

The new atomizing gun operates on compressed air at pressures above 15 pounds. The air enters at 14 (A), passes thru 13 and the quantity is controlled and released, when desired, by the mechanism 12, and passes thru 7, around the edge of 2 and out thru the tapered tube, in 1 at high velocity. Low pressure is produced at the orifice in 2 giving suction that raises the liquid to the orifice where the air at high velocity atomizes the oil solution into a fine mist or fog with considerable carrying power. The droplet size of

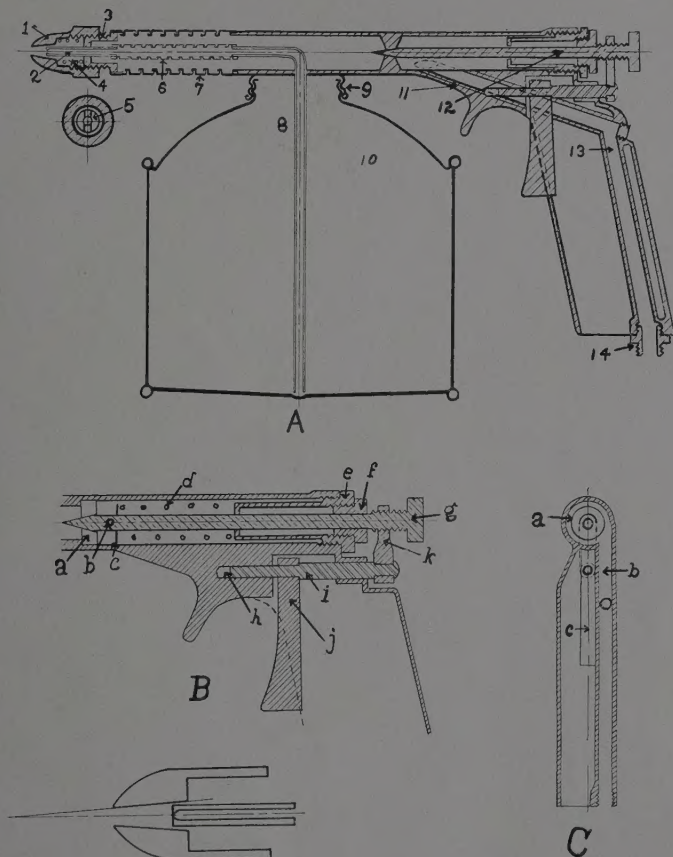


FIGURE 4. Construction of new atomizing gun.

B is a detailed drawing of 12, the air valve.

(a) Bronze valve seat driven into 11. (b) Cotter pin holding washer c. (c) Spring-retaining washer. (d) Compressing spring for closing valve. (e) Bushing (hex head) threaded to casting, drilled at closed end to allow easy movement of the valve needle. It contains packing. (f) Compression nut also drilled for the needle. (g) Needle with a knurled head threaded against the head. (h) Drilled hole in 11. (i) Shouldered steel rod. (j) Cast aluminium trigger, drilled to allow free movement of (i). It works thru a slot in 11. (k) A casting threaded for (g) at the upper end and drilled for (i) at the lower end. (g) Should work tight to hold adjustment. (k) rests against a shoulder on (i) and is firmly attached by riveting a head from the extremity of (i).

the fog produced by this device is well below that at which danger to foliage occurs. Flexible metal tubes are incorporated in the air and oil lines to facilitate spraying at different angles and the underside of foliage.

There are two main differences between the function of this new device and that of the paint-spray guns on the market. (1) This gun is designed to produce an extremely fine mist diluted with a large excess of air at high velocity to effect greater carrying properties and economy of solution, whereas paint-spray equipment gives heavy mist at low velocity with a view of applying the solution as rapidly as possible for even distribution.

(2) The principle of operation is entirely different. In this new equipment, the oil, under atmospheric pressure, is completely atomized at the orifice of the oil tube by the air at high velocity in the tapered tube. In paint spray guns, on the other hand, the liquid, under pressure, is mixed with air and the mixture forced thru a small orifice by the pressure behind it.

SUMMARY

Free nicotine dissolved in highly refined petroleum distillate shows promise as a horticultural spray. When correctly applied, it did not burn even tender plants and gave a high percent kill of many insects with piercing-sucking and chewing mouth parts.

It is concluded that 1 percent free nicotine in oil is suitable for field and greenhouse use with a minimum of discomfort to the operator, and is equivalent in toxicity to .1 per cent pyrethrum in oil.

It was found possible to dissolve free nicotine in oil by two methods. The first is by adding 100 percent free nicotine directly to the oil. The second is by shaking commercial free nicotine products, such as 50 percent water solution, with the oil and then separating the oil-nicotine from the settled water and impurities.

Nicotine in oil must be applied to plants in the form of a fine fog to avoid plant injury. Two new applicators have been devised and are described, one of which, a new hand continuous atomizer, appears to have immediate possibilities.

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APPENDIX

Table 5. Comparative toxicity of oil-nicotine and aqueous nicotine sprays.

Material	No. tests	No. insects	Percent killed
Check; oil plus interm. solvent	3	15	0
.63% free nicotine in oil	3	15	6.7
1.25% free nicotine in oil	3	15	40
2.5% free nicotine in oil	3	15	80
5% free nicotine in oil	8	40	100
2.5% free nicotine in water with spreader	3	15	0
5% free nicotine in water with spreader	3	15	0

Table 6. Toxicity tests of nicotine and other insecticidal materials in refined hydrocarbon distillate.

Bell Jar-Settling Mist				
Insect	Material	No. tests	No. alive	No. dead
Nasturtium aphid	No treatment	2	26	0
	Oil check	1	29	7
	1% oil-nicotine	3	4	84
Pea aphid	.2% oil-pyrethrum	3	17	105
	No treatment	2	23	0
	Oil check	3	64	4
	1% oil-nicotine	6	35	40
			(All paralyzed)	
	.2% oil-pyrethrum	4	30	35
Red spider			(All paralyzed)	
	.2% oil-rotenone	1	0	11
	1% oil-nicotine	4	6	101
Imported cabbage worm	.2% oil-pyrethrum	4	7	95
	Oil check	3	25	5
	1% oil-nicotine	7	56	14
	2% oil-nicotine	2	14	10
	.1% oil-pyrethrum	4	9	31
			(7 paralyzed)	
Imported Cabbage worm	.2% oil-pyrethrum	3	4	26
Hand Atomizer				
Imported Cabbage worm	1% oil-nicotine	3	8	22
	.05% oil-pyrethrum	3	1	29
Southern cabbage worm	No treatment	4	40	1
	Oil check	6	49	11
	1% oil-nicotine	6	16	45
	.05% oil-pyrethrum	6	23	37
	.1% oil-pyrethrum	6	13	47
Cabbage looper	Oil check	4	21	19
	1% oil-nicotine	4	4	36
	.05% oil-pyrethrum	4	7	33
	.1% oil-pyrethrum	4	1	39
Cabbage looper	Oil check	3	27	0
	1% oil-nicotine	3	16	13
	.1% oil-pyrethrum	3	6	18
Apple leaf hopper (nymphs)	Oil check	4	111	49
	.63% oil-nicotine	4	32	110
	1% oil-nicotine	4	6	195
Tingids	Oil check	2	21	38
	1% oil-nicotine	2	4	71
	.2% oil-pyrethrum	2	1	52
	.2% oil-rotenone	2	28	45
Mealy bug	2.5% oil-nicotine	1	0	73 adults
				22 groups of young
				49 egg masses
Potato beetle	1% oil-nicotine	5	16	8
			(6 paralyzed)	
Ham mite	No treatment	2	7	2
	Oil check	4	14	6
	1% oil-nicotine	12	41	97
	2% oil-nicotine	5	1	32
	3% oil-nicotine	2	0	21